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Single Crystal Diamond Nanomechanical Dome Resonator

M.K. Zalalutdinov,¹ J.W. Baldwin,² B.B. Pate,³ J. Yang,⁴
J.E. Butler,³ and B.H. Houston²

¹*SFA, Inc.*

²*Acoustics Division*

³*Chemistry Division*

⁴*NOVA Research, Inc.*

Introduction: Superior mechanical and optical properties of single crystal (SC) diamond in combination with a chemically inert and bio-compatible surface enable the design and fabrication of novel nanoscale devices for sensing and signal processing applications. In this letter, we present the first fabrication and operation of high-frequency nanomechanical resonators implemented in 100-nm-thick SC diamond films epitaxially grown on SC synthetic diamond. The method of fabrication is based on introducing a pattern of localized mechanical stress in the epitaxial film and can be potentially extended below 10 nm film thickness. Reduction in film thickness and hence a reduction in the mass of the resonator is motivated by a straightforward gain in performance as a mass sensor. The corresponding increase in resonant frequency greatly facilitates operation in viscous media such as air and water. The exceptional mass sensitivity of nanomechanical resonators, combined with emerging methods of functionalization¹ that provide a diamond surface with the specificity toward analytes of interest, make SC diamond resonators a superb choice for chem/bio sensing applications.

Fabrication: Prior to epitaxial film growth, the substrate (Sumitomo Electric Type-Ib, high pressure high temperature (HPHT) synthetic single crystal) was polished and implanted with carbon (energy 180 keV, dose $1 \times 10^{16} \text{ cm}^{-2}$) to form a 230-nm-deep sacrificial layer. Implantation was followed by microwave plasma-assisted chemical vapor deposition of 100-nm-thick SC diamond film² and an electrolysis-based release process³ that partially undercuts the sacrificial layer. The optical micrograph in Fig. 9 and the atomic force microscope (AFM) image in Fig. 10 show examples of the resulting structures—domes with an outer diameter below 10 microns and elevation of $\sim 200 \text{ nm}$ at the apex. We attribute the dome formation to the presence of metallic (Fe, Co, Ni) inclusions on the surface of the substrate. According to our model, the high temperature of the CVD growth (900–1150 °C) and numerous defects created by the ion implantation facilitate the diffusion of the metal atoms along the sacrificial layer, possibly accompanied with local modification of the carbon-carbon bonds. Distortion of the substrate

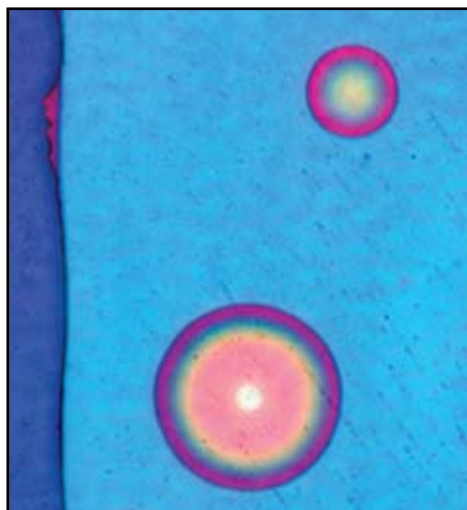


FIGURE 9

Optical micrograph of the SC diamond domes. The smaller dome is 9.2 microns in diameter.

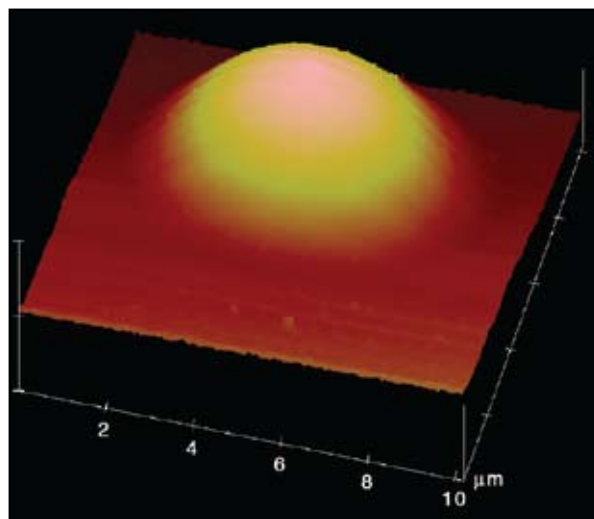
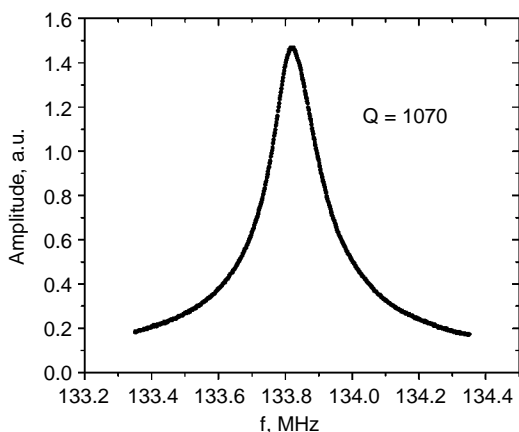


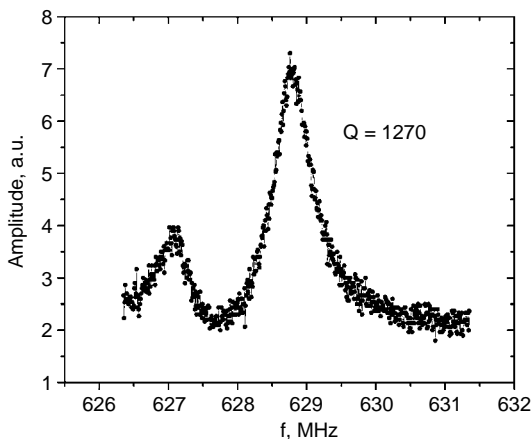
FIGURE 10

AFM image of the SC diamond dome resonator (diameter 8 microns, elevation at the apex 200 nm).

lattice contributes to stress in the CVD-grown film while the remaining particle at the center of the cloud is expected to generate a pinhole. During the electrolytic release step, the ionic transport removes the metal particle and preferentially etches the metal-enriched parts of the sacrificial layer creating a freely suspended circular membrane. Compressive stress accumulated in the CVD-grown film causes the membrane to buckle up, creating a dome-like structure. The resulting curvature of the domes provides us with an estimate for the pre-released stress in excess of 2 GPa. It should be noted that the ability to create a controlled pattern of such high stress (order of magnitude higher than in polysilicon, for example) can be exploited in numerous

**FIGURE 11**

Frequency response of the SC diamond dome resonator (7.6 microns in diameter, fundamental mode of vibration).

**FIGURE 12**

SC diamond dome resonator: overtone at 630 MHz.

microelectromechanical system (MEMS) structures that require post-release self-assembly and/or shaping, with the domes being the simplest example.

Operation: The mechanical vibrational spectrum of the dome (thin shell clamped on periphery) exhibits a series of sharp resonances corresponding to different configurations of standing flexural waves. Figure 11 shows the resonant peak at 130 MHz that corresponds to fundamental mode of vibration (γ_{00}) and demonstrates a quality factor $Q \sim 1000$ in vacuum ($P \sim 10^{-7}$ Torr). Extra rigidity added by the 3D nature of the shell is manifested by the upward shift of the resonance ($\delta f \sim 60$ MHz compared to a flat membrane of equal radius). Figure 12 shows a resonant peak at one of the overtones of the dome resonator at 630 MHz. These spectra were acquired using laser-based thermoelastic actuation and interferometric readout.⁴ A time-variable stress caused by modulated intensity of a laser beam (diode 412 nm) focused to a submicron spot within

the dome was used to activate the dome vibrations. The resulting motion was detected by monitoring the reflectivity of the second laser beam (HeNe 633 nm) focused on the dome. This optical method of interfacing nanomechanical resonators can be used to address structures enclosed in microchannels and/or exposed to a toxic environment. Further, it is implementable over a wide temperature range and most importantly, it provides the benefit of simplicity for the mechanical structure itself, thus reducing the size and the mass of the resonator. A simple estimate for the sensitivity of the SC diamond dome resonator as a mass sensor can be extracted from Eq. (1):

$$\delta m \approx \frac{M_{\text{resonator}}}{Q}. \quad (1)$$

Summary: Our SC diamond dome resonators demonstrate a quality factor $Q \sim 500$ at ambient pressure, resulting in added mass sensitivity estimated as $\delta m \sim 10^{-15}$ g. We expect to reach atto-gram (10^{-18} g) sensitivity in air with the resonators fabricated from thinner (~ 10 nm) SC diamond films, which is a sensitivity range inaccessible with polycrystalline or nanocrystalline films. The superb ruggedness of the dome resonators, their ability to withstand submersion, sonication, etc., is an enabling factor for post-processing functionalization, where different chemical agents must be selectively placed on the surface of pre-fabricated and released nanoresonators. We are currently working on the design and fabrication of large arrays of SC diamond dome resonators grown on lithographically defined nanoscale metallic seed particles in order to demonstrate sensitivity and throughput efficiency largely exceeding the performance of existing thin-film and bulk resonator-based sensors.

[Sponsored by ONR]

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